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<p>(21) International Application Number: PCT/US92/06308</p> <p>(22) International Filing Date: 30 July 1992 (30.07.92)</p> <p>(30) Priority data:</p> <table border="0"> <tr> <td>738,529</td> <td>31 July 1991 (31.07.91)</td> <td>US</td> </tr> <tr> <td>921,505</td> <td>29 July 1992 (29.07.92)</td> <td>US</td> </tr> </table> <p>(71) Applicant: THE REGENTS OF THE UNIVERSITY OF CALIFORNIA [US/US]; 300 Lakeside Drive, 22nd Floor, Oakland, CA 94612-3550 (US).</p> <p>(72) Inventor: PEREZ-MENDEZ, Victor ; 870 Santa Barbara Road, Berkeley, CA 94707 (US).</p> <p>(74) Agents: ZIMMERMAN, C., Michael et al.; Flehr, Hohbach, Test, Albritton &amp; Herbert, 4 Embarcadero Center, Suite 3400, San Francisco, CA 94111-4187 (US).</p>		738,529	31 July 1991 (31.07.91)	US	921,505	29 July 1992 (29.07.92)	US	<p>(81) Designated States: CA, JP, KR, RU, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LU, MC, NL, SE).</p> <p><b>Published</b>  <i>With international search report.</i></p>
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A cross-sectional view of a semiconductor device. The structure consists of a substrate 12 with a series of rectangular regions 13, 14, and 15 stacked vertically. A layer 16 is on top of 15, and a layer 17 is on top of 16. A series of rectangular regions 18, 19, and 20 are stacked vertically on top of 17. The regions 18, 19, and 20 are separated by gaps. The width of the regions 18, 19, and 20 is indicated by  $d_3$ . The height of the regions 18, 19, and 20 is indicated by  $d_4$ . The regions 18, 19, and 20 are connected to a common line 21, which is connected to a block labeled "READOUT ELECTRONICS". The regions 18, 19, and 20 are also connected to a common line 22, which is connected to a block labeled "READOUT ELECTRONICS".

Method and apparatus (11) for producing separated columns (19) of scintillation layer material (18), for use in detection of X-rays and high energy charged particles (23), with improved spatial resolution. A pattern of ridges or projections (16) is formed on one surface of a substrate layer or in a thin polyamide layer (15), and the scintillation layer (18) is grown at controlled temperature and growth rate on the ridge-containing material (16). The scintillation material (18) preferentially forms cylinders or columns (19), separated by gaps (20) conforming to the pattern of ridges (16), and these columns (19) direct most of the light produced in the scintillation layer (18) along individual columns (19) for subsequent detection in a photodiode layer (13). The gaps (20) may be filled with a light-absorbing material to further enhance the spatial resolution of the particle detector.

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### IMPROVEMENTS IN PARTICLE DETECTOR SPATIAL RESOLUTION

This application is a continuation-in-part of U.S. Application No. 07/738,529, filed 07/31/91. This invention was made with Government support under  
5 Contract No. DE-AC03-76SF00098 between the U.S. Department of Energy and the University of California for the operation of Lawrence Berkeley Laboratory. The Government has certain rights in this invention.

#### Field of the Invention.

- 10 This invention relates to improvements in spatial resolution in particle detectors, and more particularly to a luminescent layer in a particle detector.

#### Background of the Invention

- A luminescent material, such as cesium iodide (CsI),  
15 potassium iodide (KI), rubidium iodide (RbI), gallium selenide ( $\text{Ga}_2\text{Se}_3$ ), gadolinium oxysulphate ( $\text{Gd}_2\text{O}_2\text{S}$ ), lanthanum oxysulphate ( $\text{La}_2\text{O}_2\text{S}$ ), cadmium sulphide (CdS), zinc cadmium sulphide ( $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ ), cadmium tungstate ( $\text{CdWO}_4$ ), or lead oxide ( $\text{PbO}_2$ ), will receive incident  
20 charged particles or photons of high kinetic energy and convert part or all of this kinetic energy to one or a plurality of photons of individual energies lying in the range 1-4 eV. The electromagnetic radiation emitted by the luminescent material is not wholly  
25 directed in a single forward direction, but is emitted in all directions, although not isotropically.

Preferably, most or all of this radiation should propagate in approximately the forward direction, toward a photodiode layer that will provide an electrical signal indicating arrival of the incident high energy charged particles or photons. For this reason, many workers have attempted to promote forward direction emission of photons by the light-emitting atoms or molecules contained in the luminescent material.

- One early approach to compartmentalization of radiation produced in a luminescent layer is disclosed by MacLeod in U.S. Patent No. 3,041,456, where thin walls, running in two orthogonal directions, of optically transparent material are provided between thin adjacent layers or rectangular parallelepipeds of luminescent material.

Another early approach, disclosed by Ligtenberg et al in U.S. Patent No. 3,825,763, provides a substrate of glass or metal (e.g., Al or Ti), on which a thin scintillation layer of CsI or  $Z_x\text{Cd}_{1-x}\text{S}$  is deposited of unspecified thickness. A separation layer of  $\text{Al}_2\text{O}_3$  is deposited on an exposed surface of the scintillation layer, and a photocathode layer of  $\text{Cs}_2\text{Sb}$  or similar material is deposited on an exposed surface of the separation layer. The substrate material and the scintillation material have respective thermal expansion coefficients of  $2 - 2.5 \times 10^{-5}/^\circ\text{C}$ , respectively, and the substrate is maintained at an elevated temperature  $T = 150 - 200^\circ\text{C}$  when the scintillation material is deposited thereon. The substrate-scintillation layer combination is then cooled to room temperature, and cracks develop in the thin scintillation layer as cooling proceeds. These cracks produce columns of scintillation material, separated by small air or vacuum gaps between adjacent columns and extending approximately perpendicularly to

the substrate-scintillation layer interface. The crack structure thereby produced has a random collection of shapes and associated diameters.

5 Gudden et al, in U.S. Patent No. 3,829,378, disclose use of a luminescent layer on a screen with an absorbing substance deposited thereon whose absorption decreases as position varies from the center toward the edges of the screen. This invention partly compensates for the tendency of screen brightness to decrease as one  
10 approaches an edge of the screen.

A process for making columnar structures of a luminescent layer on an X-ray screen is disclosed in U.S. Patent No. 4,069,355 by Lubowski et al. Depressions or valleys are etched at regular intervals  
15 in an underlying substrate, and the luminescent material is grown only on the raised portions of the substrate. The gaps between adjacent columns of luminescent material are filled with a highly reflecting material or with another luminescent  
20 material.

Sonoda, in U.S. Patent No. 4,239,791, discloses a method for making a screen image intensifier. A heated phosphorescent material layer is treated with a colder liquid material, such as acetone, to cause differential  
25 thermal contraction and form a plurality of elongated cracks in this layer running approximately perpendicular to the substrate-phosphorescent layer interface. These cracks are asserted to form optically independent columns of phosphorescent material.

30 Riihimaki et al disclose an X-ray intensifying screen with a luminescent layer formed, in an unspecified manner, with a plurality of regularly spaced grooves therein to capture and guide light produced in the

luminescent layer. The grooves run in one direction only, and it is unclear how light is channelled within the air or vacuum gaps (with refractive index = 1) between the luminescent material (with refractive index  
5 > 1).

Van Leunen, in U.S. Patent No. 4,712,011, discloses use of the columnar structure produced by the Ligtenberg, et al, invention, and deposits an X-ray-absorbing material in the air/vacuum gaps to absorb X-ray light  
10 incident on a gap. Up to five percent of the weight of the scintillation layer may be X-ray-absorbing material deposited in the gaps, but no method of depositing the X-ray absorbing material is discussed.

A method for vapor deposition of a luminescent layer on  
15 a screen for image intensification is disclosed by Ligtenberg et al in U.S. Patent No. 4,842,894. The vapor deposition crucible is positioned at about 20° relative to the normal to the screen, and gaps formed between columns of the luminescent material appear to  
20 be elongated bubbles of unspecified material (possibly air or a vacuum). The luminescent material apparently forms predominantly crystalline columns of this material.

Bates, in Advances in Electronics and Electron Physics,  
25 vol. 28A (1969) pp. 451-459, discloses use of crystalline CsI and thermally-induced cracking of a contiguous substrate, with crack diameter about 0.5  $\mu\text{m}$ . It is unclear whether the subsequently grown CsI forms into spaced apart columns as a result of presence of  
30 the substrate cracks.

Stevens and Schrama-dePauw discuss some characteristics of vapor deposited CsI, activated with Na, in Philips Research Reports, vol. 29 (1974) pp. 341-352 and

- 353-362. Cracks are thermally induced in a substrate, held at a temperature of  $T = 50-300^{\circ}\text{C}$ , and a thick or thin CsI(Na) layer is subsequently grown on a cracked surface of the substrate. Stevels et al discuss the effects of heat treatment, thick versus thin CsI layers, the substrate material (KI, RbI or other) and average diameter of the cracks on columns of CsI that form on the substrate surface. The possibility of light channeling in such columns is discussed.
- 10 An X-ray image intensifier, using CsI material formed into irregular columns by a cracked mosaic pattern on a substrate, is disclosed by Washida and Sonoda in Advances in Electronics and Electron Physics, vol. 52 (1979) pp. 201-207. The possibility of X-ray channeling is discussed, and two types of column spacings are discussed. Maximum improvement of X-ray intensity, relative to use of a conventional, non-columnar luminescent material, appears to be about 40 percent.
- 20 British Patent No. 1,423,935, issued to Philips Electronics and Associated Industries, Ltd., discloses provision of a mosaic crack structure in the form of circles, hexagons and rectangles on a substrate surface. Cesium iodide or a similar luminescent material is subsequently vapor deposited on this surface. It is unclear whether spaced apart columns of this vapor deposited material form as a result of the mosaic crack structure.

30 Most or all of the work discussed above relies upon an irregular mosaic crack structure, formed by thermal mismatch or a similar process, to provide formation of columns, if any, of a deposited luminescent material. Little or no regularity or control is available for parameters such as column diameters, spacing of

adjacent columns, or the tendency of such columns to coalesce if the luminescent material is sufficiently thick. What is needed here is a method for column growth of luminescent material that provides control of these parameters and allows flexibility in formation of such structures.

#### Summary of the Invention

These needs are met by the invention, which provides one or more methods for fabricating a sequence of columns of regular, controllable geometry and diameter perpendicular to the interface of the luminescent material with an adjacent material. These columns are separated by gaps that may be evacuate or filled with air, with a light-absorbing material, or with a light-producing or light-reflecting substance. In one embodiment, a thin layer of polyimide or other suitable etchable material is deposited on a substrate structure and a sequence of ridges or projections is etched in the polyimide material in a predetermined geometrical structure. A luminescent material is slowly grown (rate  $\leq 250 \mu\text{m/hr}$ ) on the ridge-containing surface to produce a plurality of columns separated by gaps in one or two dimensions. The width of a ridge is varied according to the height of the luminescent layer to be grown so that the columns do not coalesce. Alternatively, the ridges may be etched directly into a substrate material, such as glass, plastic, metal or amorphous or crystalline silicon or germanium. In a third embodiment, the structure is put together as a "sandwich" of a first set of layers containing a photodiode layer and a second set of layers containing a luminescent layer. The columnar structure thus produced reduces by about 58 percent the optical spread factor associated with light produced by conversion in the luminescent material, in one embodiment.



Brief Description of the Drawings

Figure 1 is a sectional side view of one embodiment of apparatus constructed according to the invention.

Figures 2 and 3 are sectional side views illustrating the columns formed in the luminescent layer in Figure 1.

Figures 4, 5, 6, 7 and 8 are schematic top views of triangular, rectangular, hexagonal, ovular and linear ridge patterns constructed on an underlayer according to the invention.

Figure 9 is a sectional side view of a second embodiment of the apparatus constructed according to the invention.

Figures 10A and 10B are sectional views illustrating a third embodiment of the invention.

Figure 11 is a graphical view illustrating a measure of the improvement in spatial resolution where the invention is used for X-ray and charged particle detection.

Description of the Invention

In a first embodiment 11 of the invention, shown in Figure 1, a substrate 12 is provided on which a photodiode layer 13 is deposited on one substrate surface. The substrate material may be glass, plastic, a ceramic, a thin metal layer such as Al or Ti, or crystalline or amorphous silicon or germanium, and the photodiode material is preferably a hydrogenated amorphous column IV semiconductor material such as a-Si:H or a-Ge:H. A transparent, electrically conductive, thin layer 14 (not drawn to scale) of material such as indium-tin-oxide ("ITO"), tin-oxide ("TO"), or another suitable thin metal film is then

deposited on an exposed surface of the photodiode layer 13 with a thickness of 1-100 nanometers (nm). The conductive layer 14 is at least partly optically transparent and allows passage of electromagnetic radiation (photons) of appropriate wavelengths between a luminescent layer positioned on one side of the conductive layer and a photodiode layer 13 positioned on the other side of the conductive layer. The photodiode layer 13 and conductive layer 14 are contiguous here. The substrate layer 12 may have any appropriate thickness, and the photodiode layer 13 may have a thickness of 1-10  $\mu\text{m}$  or 10-100  $\mu\text{m}$  for amorphous semiconductor material (e.g., a-Si:H or a-Ge:H) or crystalline semiconductor material (e.g., c-Si or c-Ge), respectively. The photodiode layer 13 has a plurality of electrical traces 21 connected at one end to this layer at regular intervals, and these traces are connected at their second ends to readout electronics 22 that receives signals generated within the photodiode layer.

A pattern layer 15 of etchable pattern material 15, such as polyimide resin,  $\text{SiO}_2$ , or metals such as Al, Cr, Au, Ag, Pd or Pt, of thickness  $d = 5\text{-}20\ \mu\text{m}$ , is then deposited on an exposed surface of the conductive layer 14 as shown. Portions of the pattern layer 15 are etched to produce a one-dimensional or two-dimensional sequence of regularly spaced ridges 16 of the pattern material that project approximately perpendicularly to the interface or surface 17 between the conductive layer 14 and the pattern layer. The ridges 16 have height  $\approx d_1$ , lateral thickness or width  $\approx d_2$ , and are spaced apart from adjacent ridges by a distance  $\approx d_3$ . In a preferred embodiment, these lengths are  $d_1 = 5\text{-}20\ \mu\text{m}$ ,  $d_2 = 2\text{-}20\ \mu\text{m}$ , and  $d_3 = 10\text{-}100\ \mu\text{m}$ . The "pitch" of this pattern of ridges,  $p = d_2 + d_3$ , may vary from 15  $\mu\text{m}$  to 110  $\mu\text{m}$ . In the etching of the etchable pattern

material 15, a small portion of this material may be allowed to remain at the interface 17, as shown in Figure 1; or the etchable pattern material 15 may be removed down to the interface 17, except for the ridges 16. In this latter situation, the interface 17 might be covered with a thin layer (not shown in Figure 1) of an etch stop material, such as a native oxide or silicon nitride, in a manner well known to workers in this art.

10 The structure is then heated to a low temperature  $T = 50 - 400^{\circ}\text{C}$  (preferably,  $T = 100-250^{\circ}\text{C}$ ), and a luminescent layer 18 of luminescent material, such as CsI, KI, RbI, CdS,  $\text{CdWO}_3$ ,  $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ ,  $\text{Ga}_x\text{Se}$ ,  $\text{Gd}_2\text{O}_3\text{S}$ ,  $\text{La}_2\text{O}_3\text{S}$ ,  $\text{PbO}_2$  or other suitable luminescent material, is grown  
15 slowly by evaporation at a growth rate  $\leq 500 \mu\text{m/hr}$  (preferably  $\leq 250 \mu\text{m/hr}$ ) over the interface 17 and ridges 16, to a height  $d_4 \approx 100-1,000 \mu\text{m}$ . This evaporation process produces a sequence of cylinders or columns 19 of the luminescent material, separated by  
20 air or vacuum gaps 20 of diameter  $d_{\text{gap}} < d_2$  because of the presence of the ridges 16 of lateral thickness  $d_2$ . Use of a low growth rate improves adhesion and light emission efficiency of the luminescent material. Certain of the luminescent materials may be activated  
25 with Na (CsI), with Tl (CsI), or with a rare earth such as Ce, Pr, Nd or one of the other 11 rare earth elements ( $\text{Gd}_2\text{O}_3\text{S}$ ).

If the thickness  $d_4$  of the luminescent layer 18 is made sufficiently large, it is possible that adjacent  
30 columns 19 will coalesce in an upper part of the layer 18, although the columns 19 will be well separated from one another in the lower part of this structure. This is illustrated schematically in Figure 2. However, if the luminescent layer thickness  $d_4$  is kept sufficiently

small (say,  $d_4 \leq 1,000 \mu\text{m}$ ) and the evaporation rate for the luminescent layer 18 is kept small enough, the resulting luminescent layer should consist of columns 19 of luminescent material that are completely  
5 separated from one another. For example, a ridge that is  $d_2 = 8 \mu\text{m}$  wide will produce a gap between two adjacent columns of CsI material that does not close until the heights of the two adjacent columns reaches about  $450 \mu\text{m}$ . This effect scales approximately  
10 linearly with width  $d_2$  of the ridge. Thus, if ridges of lateral width  $d_2$  are grown, the thickness  $d_4$  of the luminescent layer should preferably be no more than  $50 d_2$  in order to avoid gap closure. However, luminescent layers of thicknesses  $d_4 = 70 d_2$ , or even  $d_4 = 100 d_2$ ,  
15 prepared according to the invention, will provide improved spatial resolution for particle detection. Thus, a ridge of width  $d_2 = 20 \mu\text{m}$  is predicted to produce a gap between two adjacent columns of CsI that does not close for a column height  $d_4 = 1,000 \mu\text{m}$ . The  
20 other luminescent materials mentioned above are believed to behave qualitatively similarly so that the minimum height required for gap closure scales approximately linearly with ridge width  $d_2$ .

As illustrated in Figure 3, energetic particles 23  
25 (high energy photons or massive charged particles) that are incident upon the embodiment 11 (Figure 1) first encounter the luminescent layer 18, and all or a portion of the kinetic energy of these particles is converted to electromagnetic radiation or photons 25,  
30 of wavelength  $\lambda_0$  lying in the range  $0.3 \mu\text{m} \leq \lambda_0 \leq 0.7 \mu\text{m}$ , emitted by the luminescent material. A photon 25 will propagate generally toward the interface 17 (Figure 1) and will encounter and/or be reflected from the wall of the column in which that photon was  
35 originally produced. If the incidence angle  $\theta$  of the

photon 23 relative to a local tangent to the column wall is sufficiently large ( $\csc\theta \geq \csc\theta_{cr} = n_L =$  refractive index of luminescent material at that wavelength), the photon 25 will undergo total internal reflection and will remain within and propagate within the column in which the photon was originally produced by kinetic energy conversion. If the conversion radiation is assumed to be emitted isotropically by the luminescent material, the trapped fraction  $f_T$  of this emitted radiation that undergoes total internal reflection in the forward direction from a column wall is determined to be

$$f_T = \int_0^{\theta_{cr}} 2\pi \sin\theta \, d\theta / \int_0^{\pi} 2\pi \sin\theta \, d\theta = (1 - 1/n_L)/2. \quad (1)$$

Table 1 presents the refractive indices and trapped fractions  $f_T$  for some luminescent materials of interest. Although the fraction  $f_T$  is less than 0.5, the fact that much of this radiation stays within the column in which it is produced enhances the spatial resolution of the particle detector constructed according to the embodiment 11 in Figure 1. This trapped fraction  $f_T$  is approximately independent of the height of a column.

25 Table 1. Refractive Index and Trapping Fraction for Luminescent Material Columns

	<u>Luminescent Material</u>	<u>Refractive Index</u>	<u><math>f_T</math></u>
	CsI	1.78	0.22
30	KI	1.68	0.20
	RbI	1.65	0.20
	CdS	2.51	0.30
	$Zn_xCd_{1-x}S$	2.36 (est.)	0.29
	$La_2O_2S$	1.56	0.18
35	$PbO_2$	2.3 (est.)	0.18

Figures 4, 5, 6, 7 and 8 are schematic top views of triangular, rectangular, hexagonal, ovular (or circular) and "linear" arrays that provide suitable one-dimensional and two-dimensional ridge patterns for the etchable material 15 used in the embodiment 11 in Figure 1. The linear array 51 shown in Figure 8 produces a sequence of parallel blocks or columns extending above the regions 55 and separated by gaps defined by the ridges 53. More generally, an array of closed polygonal ridges may be provided to define and promote initial formation of the columns and separating gaps.

Figure 9 illustrates a second embodiment 81 of the invention, in which the order of some of the layers is reversed. A substrate layer 83 is patterned and etched to provide a sequence of ridges 85 of the substrate material extending approximately perpendicularly to the adjacent surface of the substrate. As before, the substrate layer material may be glass, plastic, thin metal, or crystalline or amorphous silicon or germanium. Alternatively, a thin polyimide layer 84 may be deposited on the exposed surface of the substrate 83 and used for ridge formation by etching. A luminescent layer 87, containing luminescent material such as CsI, KI, RbI, CdS,  $\text{ZnxCd}_{1-x}\text{S}$ ,  $\text{CdWO}_3$ ,  $\text{Ga}_x\text{Se}$ ,  $\text{Gd}_2\text{O}_2\text{S}$ ,  $\text{La}_2\text{O}_2\text{S}$  or  $\text{PbO}_2$ , with or without any of the activators mentioned above, is then slowly evaporated over the substrate 83 patterned with the ridges 85, with the substrate temperature T being held relatively low at  $T = 100\text{--}250^\circ\text{C}$ . The luminescent material forms into a plurality of columns 89 that are separated by a sequence of air or vacuum gaps 91 defined by the ridges 85 as in Figure 1. A sealant layer 93 of polyimide or similar materials is deposited over the luminescent layer 87, and an optically transparent conductive layer 95 of ITO or TO is then deposited over the sealant

layer 93. Finally, a photodiode layer 97 of a-Si:H, a-Ge:H or mixture thereof is deposited over the conductive layer. The dimensions  $d_1$ ,  $d_2$ ,  $d_3$  and  $d_4$  of the ridges 85 and luminescent layer 87 are as in Figure 1. The sealant layer 93, the conductive layer 95 and the photodiode layer 97 have preferred thicknesses in the respective ranges 5-10  $\mu\text{m}$ , 1-100 nm, and 1-10  $\mu\text{m}$  (or 10-100  $\mu\text{m}$ ), respectively. Energetic particles 99 are incident upon the structure 81 and will pass through the luminescent layer 87 before passing through the photodiode layer 97.

Figure 10A illustrates a third embodiment of the invention, which is initially constructed as two separate sets of layers. The first set of layers includes a substrate 101, of arbitrary thickness, with a photodiode layer 103 of a-Si:H, a-Ge:H (or a combination thereof), c-Si or c-Ge deposited on the first substrate 101. As in the other two embodiments, the thickness of the photodiode layer may be 1-10  $\mu\text{m}$  for amorphous Si or Ge and 10-100  $\mu\text{m}$  for crystalline Si or Ge. The second set of layers includes a second substrate 105, on which is deposited a luminescent layer including a plurality of cylinders or columns 107 of luminescent material, such as CsI, KI, RbI, CdS,  $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ ,  $\text{CdWO}_3$ ,  $\text{Ga}_x\text{Se}$ ,  $\text{Gd}_2\text{O}_2\text{S}$ ,  $\text{La}_2\text{O}_2\text{S}$  or  $\text{PbO}_2$ . The columns 107 are separated by a plurality of one-dimensional or two-dimensional gaps 109 that are initially formed using ridges 111. The ridges 111 are in turn formed as in the first or second embodiments, by etching polyimide or another suitable etchable material or by etching the underlying substrate 105. In Figure 10B, the first and second set of layers are brought together as a "sandwich" and are, optionally, held together using a suitable "glue", such as optical grease, Canadian balsam or other suitable material that produces no effluents when this material cures and

hardens. The assembled apparatus, as shown in Figure 10B with the associated electronics 113, then functions as a particle detector with improved spatial resolution. One advantage of this third embodiment is that the first and second sets of layers may be fabricated independently. If a high temperature is needed to fabricate the columnar luminescent layer 107, the photodiode layer 103 is fabricated separately and is not subjected to this high temperature. Readout electronics is also present, but not shown, in Figures 2, 3, 9 and 10B.

Example. The following procedure is preferred for producing a ridge pattern in a polyimide layer. The substrate used should be rinsed in de-ionized water at a temperature of  $T = 80^{\circ}\text{C}$  for a time interval  $\Delta t = 3 - 10$  minutes. Polyimide material, such as DuPont PI 2555, is spun on an exposed surface of the substrate at a rotation rate of about 3500 rpm for a time interval  $\Delta t = 30$  sec, or for a time sufficient to produce a polyimide layer of an approximate thickness of  $3.5 \mu\text{m}$ . Alternatively, DuPont PI 2722 material, which contains both the polyimide and a photoresist material, may be used in place of the PI 2555 material. The substrate-polyimide layer combination is then softbaked for about 3 min at a temperature  $T = 90^{\circ}\text{C}$ . Photoresist, such as KTI 820, is then spun on top of the polyimide layer at a rotation speed of about 3500 rpm for a time interval  $\Delta t = 30$  sec, or for a time sufficient to produce a photoresist thickness of  $2.5 - 3.4 \mu\text{m}$ . The substrate-polyimide layer-photoresist layer is then softbaked for 2 min. at a temperature  $T = 120^{\circ}\text{C}$ . The photoresist is then exposed to ultraviolet radiation in the desired pattern for about 15 sec, using photolithography equipment and procedures known in the art. The irradiated photoresist is then baked for about 60 sec. and is developed. The



photoresist is immersed in a suitable developer, such as Kodak 934, for about 120 sec., then rinsed and dried. The polyimide is etched simultaneously when the photoresist is developed. The resulting ridge structure is then hardbaked or cured for at least one hour at a temperature of  $T = 280^{\circ}\text{C}$ , or for one half hour at  $T = 350^{\circ}\text{C}$ . The scintillation layer material is then coated on the ridge structure to the desired thickness  $d_4 = 100 - 1,000 \mu\text{m}$ , using a low evaporation rate to allow columns of scintillation material to form in conformity to the ridge pattern provided.

Figure 11 is a graphical view illustrating the improvement in spatial resolution, using the invention in one embodiment. The solid circles represent intensity of light received through a conventional, unstructured layer of CsI of thickness  $450 \mu\text{m}$  with an a-Si:H photodiode layer and have an associated FWHM of  $540 \mu\text{m}$  about a selected central position at  $x = 1,500 \mu\text{m}$ . The triangles represent intensity of light received through a columnar structure of  $450 \mu\text{m}$  thick CsI, fabricated according to the invention, with an a-Si:H photodiode layer in the same configuration. Here, the FWHM is  $230 \mu\text{m}$ , a reduction of about 58 percent in the point spread width in one dimension; in two dimensions, the point spread width reduction is estimated to be about 72 percent. A particle detector constructed using the invention disclosed here may resolve lateral spatial separations as small as  $10 - 50 \mu\text{m}$ .

The gaps 20 or 91 or 109 in Figures 1, 2, 9 or 10A/10B may be filled, by capillary action or otherwise, with a light-absorbing material, such as Te, Sb or Sn (useful for CsI), to reduce the point spread factor associated with light produced in a luminescent layer column fabricated according to the invention. Using

this embodiment, most of the light that is not internally reflected within a given column is absorbed by the material in an adjacent gap and does not appear in another such column. This will reduce the total amount of light produced and transmitted by the luminescent layer but will improve the spatial resolution by ensuring that substantially all light produced in a given column of luminescent material will either be absorbed or will propagate within that column to the photodiode layer, where associated readout electronics 22 (Figure 1) detects which column of luminescent material produced the material. The absorbing material used here should have an optical absorption edge that reaches its maximum near, but below, the range of wavelengths produced by the luminescent material.

It is advantageous in the inventive method to provide interstitial material in the gaps between the cylinders or column structures which comprise the scintillation layer of the inventive detector. Spatial resolution can be improved by from 10% to 30% using this approach. The interstitial material serves a variety of functions, including increasing structural integrity of the scintillation layer and improving the sensitivity of the detection capability.

Interstitial material increases the sensitivity of detection by limiting the intercolumnar transfer of light, particles, heat, and other factors. For instance, low energy X-rays are typically generated in the columns. These will often be emitted from the column wall, generally at an incident angle. When this occurs, the low energy X-rays commonly impact adjacent columns, inducing cross talk in the detector of the adjacent column. This background signal lessens the acuity of the resulting detector, and compromises its

acuity of the resulting detector, and compromises its spatial resolution.

Scintillation layer light generation which occurs at the periphery of the columnar surface or communicates to the interstitial areas also is a source of background noise. For instance, light which escapes internal reflection may enter an adjacent column, or even scatter farther afield of the actual point of production. As such, it will give a false reading as to the spatial position of the particle impact.

The ill effects of random light scatter and detection of errant low energy X-rays can be limited or fully ameliorated by the use of interstitial materials. The choice of the material depends on a compromise between the desired results and ease of application. For instance, a multipurpose material would absorb both light and X-rays. Even if such a material did not have optimum qualities in both these functions, if it can perform both to a reasonable level it may be the material of choice. Examples of such multipurpose materials are  $Tl_2O$  and  $WCl_6$ .

For light attenuation, it is useful to provide an interstitial material that absorbs light in the range of 300-700 nm in wave length. Alternatively, the interstitial material may be reflective in nature. This has the advantage of returning the light signal to the appropriate column. The use of such materials allows the possibility of the light being correctly redirected through the column thus preventing a reading error.

Appropriate candidates for light absorbing interstitial materials are various dyes and inks, Dykem Blue,

mercury and its salts, metals and their alloys, iodine and the like.

Interstitial material should have the capacity to absorb low energy X-rays produced within columns and capable of reaching adjacent columns. This quality is exemplified by a high density and a high Z value, preferably above 50. Mercury has high Z value and high density, and so is an excellent absorber of errant low energy X-rays. Mercury salts would have similar capacities. At a Z value of 50, iodine is in an acceptable range of X-ray absorption capability.

Various methods for depositing the desired material in the interstitial areas of the scintillation layer are available. Two useful methods are vapor deposition and application through a solute carrier. Common objectives of these two methods are to achieve uniformity of layer deposition, full penetration of the interstitial area, and excellent adhesion of the layer to the substrate.

Vapor deposition must occur at a temperature which will not damage various components of the detector. In one example of the present invention, the cesium iodide layer is the limiting material in this regard. Therefore, materials selected for evaporation deposition in this case must have an evaporation temperature of less than 650 degrees Celsius. Good candidates for this process are thus mercury, with an evaporation temperature of 365 degrees Celsius, and iodine, with an evaporation temperature of less than 200 degrees Celsius. Various organic and inorganic compounds will also be useful in the vapor deposition process.

Vapor deposition is accomplished by confining the scintillator and the interstitial material within an enclosed vessel. The interstitial material is then evaporated, and recondensed on the substrate surface and interstitial area. In most cases, the material is heated to provide the necessary evaporation. The substrate is preferably heated to increase wetting and creep effects, and to improve adherence. A higher quality deposition in a shorter period of time is achieved compared to a system where the substrate is not heated. Typically, the entire treatment chamber is warmed to avoid preferential deposition on the chamber wall.

As a rule, the more gradual the heating and subsequent cooling, the finer the quality of the resultant deposited layer. Placing the substrate towards the top of the chamber avoids droplets of precipitating interstitial material should the chamber become supersaturated. One wishes to avoid the formation of liquid on the surface of the substrate prior to the filling of the interstitial areas. Processing parameters can be modified to assure that the condensation occurs uniformly within the columnar interstitial regions.

The interstitial area can also be filled using materials dissolved or suspended in a liquid carrier. In the case of the presently described materials, water as a solvent is counter-indicated because of its ill effect on the inventive materials. Useful solvents in this regard are non-aqueous materials such as acetone, carbon tetrachloride, anhydrous alcohols and the like.

While a solution close to saturation with the material to be deposited is generally preferred, this consideration must be balanced by the ill effects of a

viscous solution. Some amount of heating is possible to alleviate this problem.

Appropriate solutions for treatment of the inventive scintillation layers are iodine dissolved in anhydrous methyl or higher alcohols, or commercial dye materials such as organic inks. Sequential dipping and five minute drying produces an excellent result with these materials.

A required finishing step is common to both the vapor deposition and the solution deposition processes. If filling of the interstices to the surface level is to be assured, deposition must also be allowed on the surface. However, in the functioning detector, this layer must be removed. There are many methods available for removing this surface coating, including chemical dissolution and mechanical abrasion.

Solvent removal of the surface film must be approached with care to avoid damage of the interstitial deposits. Typically, the solvent is provided in an absorbent material, such as a blotter, and wiped across the surface. This is done only until visual observation indicates removal of the undesired film.

Mechanical abrasion can also be used to remove the excess film layer. Simple sanding or scraping will often surface for the needs of the present invention. This may have an advantageous effect of preparing the surface for application of additional components.

Claims

1. A method of producing a detector of X-rays and high energy charged particles with improved spatial resolution, the method comprising the steps of:

5 providing a substrate layer of material with an electrically conductive surface;

forming a particle detector layer containing a-Si:H, a-Ge:H, or other appropriate amorphous semiconductor material on the conductive surface of the substrate layer;

10 forming a conductive layer of electrically conductive material that is at least partly transparent to incident light having a wavelength  $\lambda$  in the range  $0.3 \mu\text{m} \leq \lambda \leq 0.7 \mu\text{m}$ , adjacent to the particle detector layer so that the particle detector layer lies between the substrate layer and the conductive layer;

forming a pattern formation layer, having at least a predetermined minimum thickness and containing an etchable material, adjacent to the conductive layer so that the conductive layer lies between the particle detector layer and the pattern formation layer;

25 etching a predetermined pattern in the pattern formation layer so that the etchable material forms a plurality of ridges or projections, of lateral width  $d$ , on an exposed surface of the pattern formation layer; and

forming a scintillation layer, at a growth rate  $\leq 500 \mu\text{m}$  per hour, at a temperature  $T$  in the range  $50^\circ\text{C} \leq T \leq 400^\circ\text{C}$ , and of thickness no more than  $100 d$ , of scintillation material that produces light of wavelength in the range  $0.3 - 0.7 \mu\text{m}$  in response to receipt of high energy particles thereat,

whereby the scintillation material forms as cylinders or columns, separated by gaps that conform to the predetermined pattern.

2. The method of claim 1, further comprising the step of choosing said scintillation layer material from the class consisting of CsI, KI, RbI, CdS,  $\text{Zn}_x\text{Cd}_{1-x}\text{S}$ ,  $\text{Gd}_2\text{O}_3\text{S}$ ,  $\text{La}_2\text{O}_3\text{S}$ ,  $\text{Ga}_x\text{Se}$ ,  $\text{CdWO}_4$  and  $\text{PbO}_2$ .
- 5 3. The method of claim 1, further comprising the step of choosing said conductive layer material from the class consisting of ITO, TO, Al, Cr, Au, Ag, Pd and Pt.
- 10 4. The method of claim 1, further comprising the step of choosing said pattern formation layer material from the class consisting of polyimide, amorphous silicon, crystalline silicon, amorphous germanium, crystalline germanium, or metals such as Al, Cr, Au, Ag, Pt and Pd..
- 15 5. The method of claim 4, further comprising the step of choosing said predetermined pattern for said pattern formation layer from the class consisting of a triangle, a quadrilateral, a hexagon, an oval and a linear array pattern.
- 20 6. The method of claim 1, further comprising the step of filling said gaps between said columns with a material.
7. The method of claim 1, further comprising the step of choosing said scintillation layer growth rate to be
- 25 no more than  $250\text{ }\mu\text{m}$  per hour.
8. The method of claim 1, further comprising the step of choosing said temperature  $T$  to lie in the range  $100^\circ\text{C} \leq T \leq 250^\circ\text{C}$ .



9. The method of claim 1, further comprising the step of choosing said thickness of said scintillation layer to be no more than 50 d.

10. The method of claim 1, further comprising the step  
5 of choosing said lateral width d to be in the range 2-20  $\mu\text{m}$ .

11. A method of producing a detector of X-rays and high energy charged particles with improved spatial resolution, the method comprising the steps of:

10 providing a substrate layer of material having an electrically conductive surface and containing an etchable material;

etching a predetermined pattern in the conductive surface of the substrate so that the etchable material  
15 forms a plurality of projections of lateral width d on the conductive surface of the substrate surface;

forming a scintillation layer, at a growth rate  $\leq 500 \mu\text{m}$  per hour, at a temperature T in the range  $50^\circ\text{C} \leq T \leq 400^\circ\text{C}$ , and of thickness no more than 100 d, of  
20 scintillation material that produces light of wavelength  $\lambda$  in the range  $0.3 \mu\text{m} \leq \lambda \leq 0.7 \mu\text{m}$  in response to receipt of high energy particles thereat;

forming a thin layer of sealant material on an exposed surface of the scintillation layer so that the  
25 scintillation layer lies between the substrate layer and the etchable material layer;

forming a conductive layer of electrically conductive material that is at least partly transparent to incident light having a wavelength lying in the  
30 range  $0.3 \mu\text{m} \leq \lambda \leq 0.7 \mu\text{m}$ , adjacent to an exposed surface of the sealant material layer so that the sealant material layer lies between the scintillation layer and the conductive layer;

forming a particle detection layer adjacent to an  
35 exposed surface of the conductive layer and containing

a-Si:H, a-Ge:H, or other appropriate amorphous semiconductor material;

whereby the scintillation material forms as cylinders or columns, separated by gaps that conform to the predetermined pattern.

12. The method of claim 11, further comprising the step of choosing said scintillation layer material from the class consisting of CsI, KI, RbI, CdS,  $Zn_xCd_{1-x}S$ ,  $Gd_2O_2S$ ,  $La_2O_2S$ ,  $Ga_2Se$ ,  $CdWO_3$ , and  $PbO_z$ .

10 13. The method of claim 11, further comprising the step of choosing said conductive layer material from the class consisting of ITO, TO, Al, Cr, Au, Ag, Pd and Pt.

15 14. The method of claim 11, further comprising the step of choosing said pattern formation layer material from the class consisting of polyimide, amorphous silicon, crystalline silicon, amorphous germanium, crystalline germanium, or metals such as Al, Cr, Au, Ag, Pt or Pd.

20 15. The method of claim 14, further comprising the step of choosing said predetermined pattern for said pattern formation layer from the class consisting of a triangle, a quadrilateral, a hexagon, an oval and a linear array pattern.

25 16. The method of claim 11, further comprising the step of filling said gaps between said columns with a material.

18. The method of claim 11, further comprising the step of choosing said temperature T to lie in the range  
30  $100^\circ C \leq T \leq 250^\circ C$ .

19. The method of claim 11, further comprising the step of choosing said thickness of said scintillation layer to be no more than 50 d.

20. The method of claim 11, further comprising the step of choosing said lateral width d to be in the range 2-20  $\mu\text{m}$ .

21. A method of producing a detector of X-rays and high energy charged particles with improved spatial resolution, the method comprising the steps of:

10 forming a first structural component consisting of a first substrate layer of material and a particle detector layer, containing a-Si:H, a-Ge:H, or other appropriate amorphous semiconductor material and deposited on an exposed surface of the first substrate layer;

15 forming a second structural component by:

forming a second substrate layer of material containing an etchable material on an exposed surface;

20 etching a predetermined pattern in the etchable material of the second substrate layer to form a plurality of ridges or projections, of lateral width d, on an exposed surface of the etchable material; and

forming a scintillation layer, at a growth rate  $\leq 500 \mu\text{m}$  per hour, at a temperature T in the range 25  $50^\circ\text{C} \leq T \leq 400^\circ\text{C}$ , and of thickness no more than 100 d, of scintillation material that produces light having a wavelength  $\lambda$  lying in the range  $0.3 \mu\text{m} \leq \lambda \leq 0.7 \mu\text{m}$  in response to receipt of high energy particles, so that the scintillation material forms as cylinders or 30 columns, separated by gaps that conform to the predetermined pattern; and

bringing the first and second structural components together so that an exposed surface of the particle detection layer is adjacent to an exposed 35 surface of the scintillation layer.

22. The method of claim 21, further comprising the step of providing an optically transparent material, positioned between said scintillation layer and said particle detection layer, to hold said scintillation  
5 layer and said particle detector layer together.

23. The method of claim 21, further comprising the step of choosing said scintillation layer material from the class consisting of CsI, KI, RbI, CdS,  $Zn_xCd_{1-x}S$ ,  $Gd_2O_2S$ ,  $La_2O_2S$ ,  $Ga_ySe$ ,  $CdWO_3$ , and  $PbO_z$ .

10 24. The method of claim 21, further comprising the step of choosing said pattern formation layer material from the class consisting of polyimide, amorphous silicon, crystalline silicon, amorphous germanium, crystalline germanium, or metals such as Al, Cr. Au,  
15 Ag, Pt, or Pd.

25. The method of claim 21, further comprising the step of choosing said predetermined pattern for said pattern formation layer from the class consisting of a triangle, a quadrilateral, a hexagon, an oval and a  
20 linear array pattern.

26. The method of claim 21, further comprising the step of filling said gaps between said columns with a material.

27. The method of claim 21, further comprising the  
25 step of choosing said scintillation layer growth rate to be no more than  $250 \mu m$  per hour.

28. The method of claim 21, further comprising the step of choosing said temperature  $T$  to lie in the range  $100^\circ C \leq T \leq 250^\circ C$ .

29. The method of claim 21, further comprising the step of choosing said thickness of said scintillation layer to be no more than 50 d.

30. The method of claim 21, further comprising the step of choosing said lateral width d to be in the range 2-20  $\mu\text{m}$ .

31. Apparatus for detection of X-rays and high energy charged particles with improved spatial resolution of detection, the apparatus comprising:

- 10 a first substrate, having an electrically conductive surface;
- a particle detector layer, formed on the conductive surface of the first substrate;
- an electrically conductive layer, formed on an exposed surface of the particle detector layer;
- 15 a scintillation layer, formed on an exposed surface of the conductive layer as a plurality of columns of scintillation material that produces light having a wavelength  $\lambda$  lying in a range  $0.3 \mu\text{m} \leq \lambda \leq 0.7 \mu\text{m}$  in response to receipt of high energy particles, with adjacent columns of scintillation material being separated by gaps of maximum lateral width d and the columns of scintillation material having a height no more than 100 d; and
- 25 a sealant layer, formed over an exposed surface of the scintillation layer, where the gaps between the columns of luminescent material conform to a predetermined pattern at the interface between the conductive layer and the luminescent layer, or at the interface between the sealant layer and the luminescent layer.
- 30

32. The apparatus of claim 31, wherein said scintillation layer material is drawn from the class

consisting of CsI, KI, RbI, CdS,  $Zn_xCd_{1-x}S$ ,  $Gd_2O_3S$ ,  $La_2O_3S$ ,  $Ga_3Se$ ,  $CdWO_3$ , and  $PbO_x$ .

33. The apparatus of claim 31, wherein said predetermined pattern is drawn from the class  
5 consisting of a triangle, a quadrilateral, a hexagon, an oval and a linear array pattern.

34. The apparatus of claim 31, wherein said height of said columns of said scintillation material is no more than 50 d.

10 35. The method of Claim 6, wherein the material absorbs or reflects light and/or subatomic particles.

36. The method of Claim 35, wherein the material absorbs light in the range of 300-700 nm.

15 37. The method of Claim 35, wherein the material is dense and has a high Z value.

38. The method of Claim 35, wherein the material is selected from the group comprising dyes, inks, metals and their alloys, organic compounds and inorganic compounds.

20 39. The method of Claim 38, wherein the material is selected from the group comprising Dykem Blue, iodine,  $TlO_2$ ,  $WCl_6$ , and mercury and its salts.

40. The method of Claim 6, wherein the material is deposited using vapor deposition.

25 41. The method of Claim 6, wherein the material is deposited in a solute.

42. The method of Claim 16, wherein the material absorbs or reflects light and/or subatomic particles.

43. The method of Claim 42, wherein the material absorbs light in the range of 300-700 nm.

5 44. The method of Claim 42, wherein the material is dense and has a high Z value.

45. The method of Claim 42, wherein the material is selected from the group comprising dyes, inks, metals and their alloys, organic compounds and inorganic  
10 compounds.

46. The method of Claim 45, wherein the material is selected from the group comprising Dykem Blue, iodine,  $TlO_2$ ,  $WCl_6$ , and mercury and its salts.

47. The method of Claim 16, wherein the material is  
15 deposited using vapor deposition.

48. The method of Claim 16, wherein the material is deposited in a solute.

49. The method of Claim 26, wherein the material absorbs or reflects light and/or subatomic particles.

20 50. The method of Claim 49, wherein the material absorbs light in the range of 300-700 nm.

51. The method of Claim 49, wherein the material is dense and has a high Z value.

52. The method of Claim 49, wherein the material is  
25 selected from the group comprising dyes, inks, metals and their alloys, organic and inorganic compounds.

53. The method of Claim 52, wherein the material is selected from the group comprising Dykem Blue, iodine,  $TlO_2$ ,  $WCl_6$ , and mercury and its salts.

54. The method of Claim 26, wherein the material is  
5 deposited using vapor deposition.

55. The method of Claim 26, wherein the material is deposited in a solute.





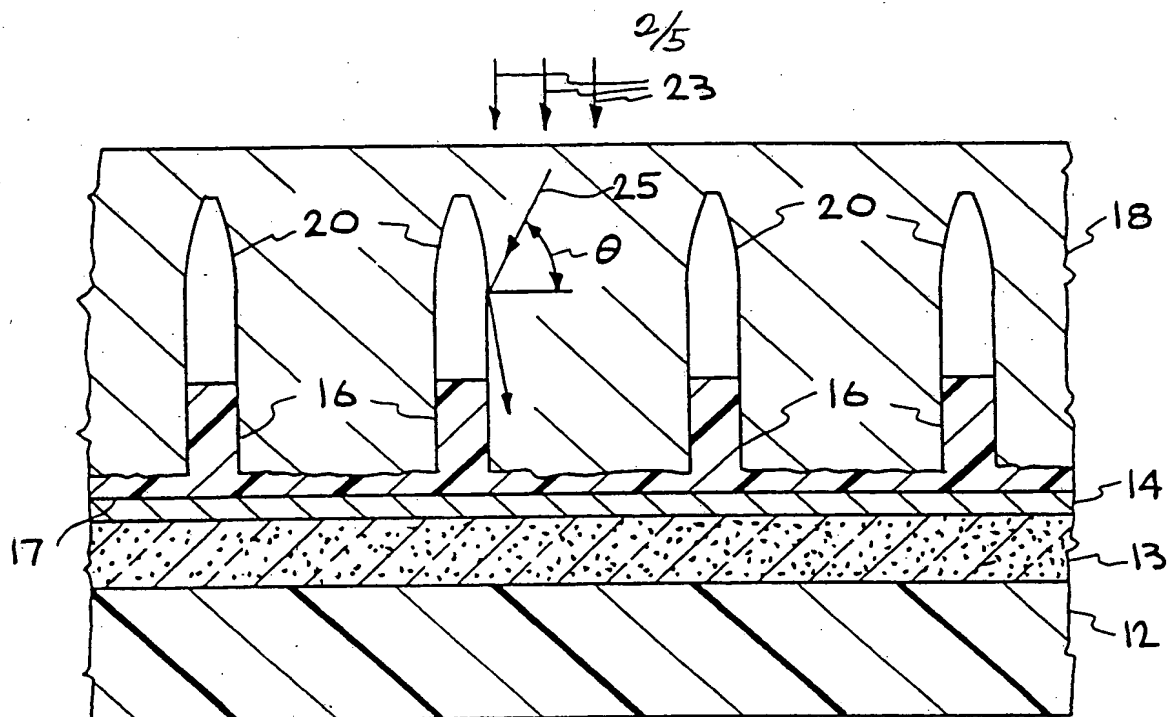


FIG. 3

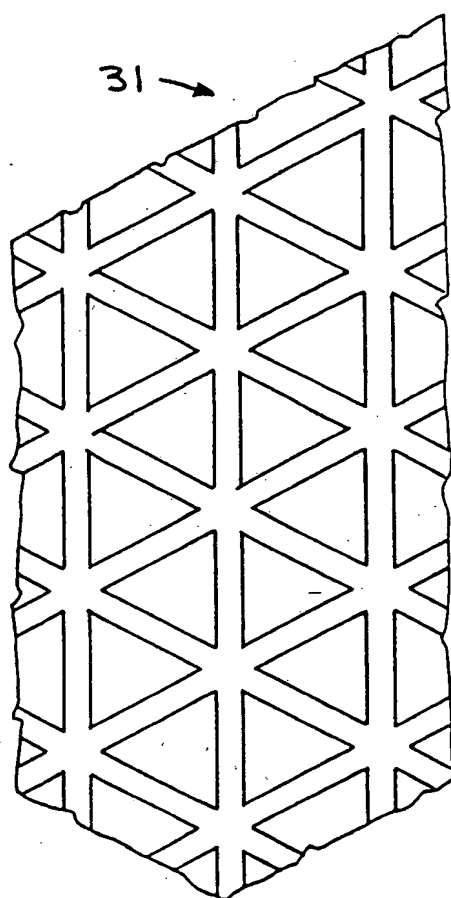


FIG. 4

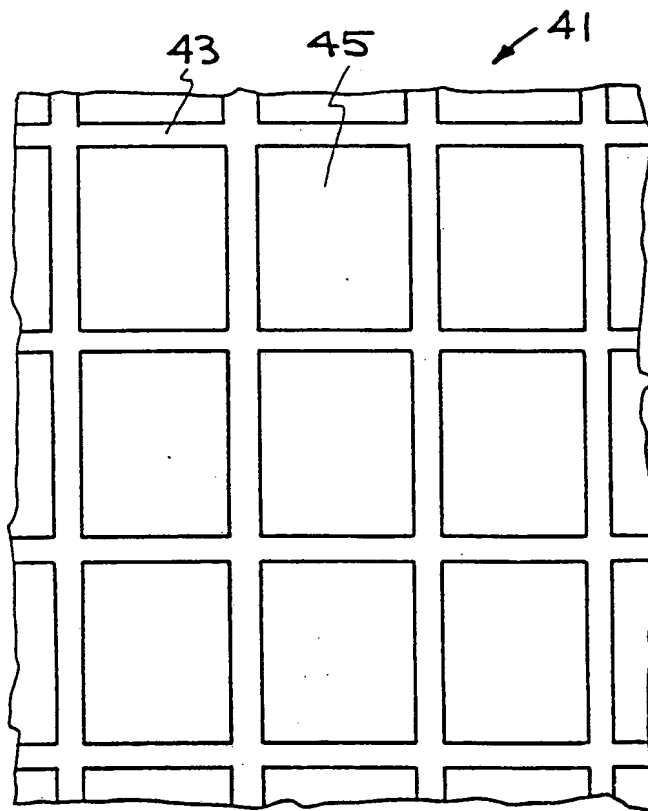


FIG. 5

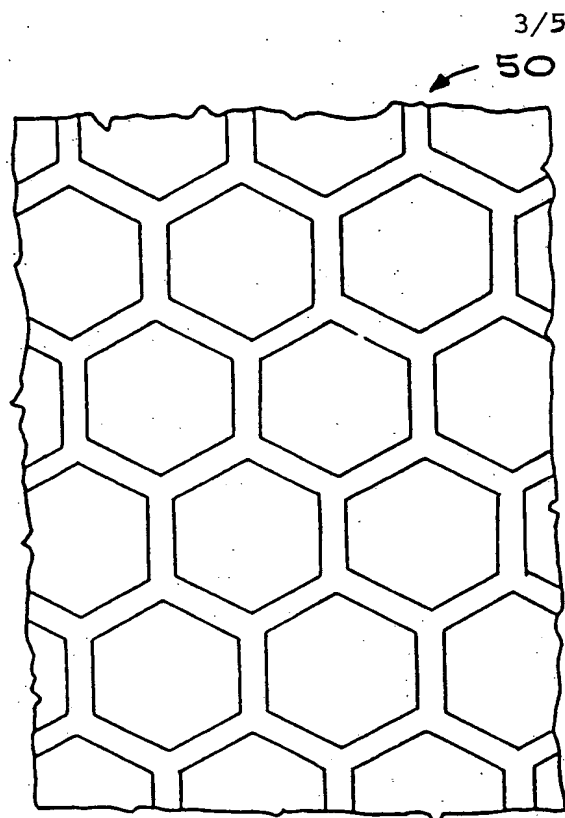


FIG. 6

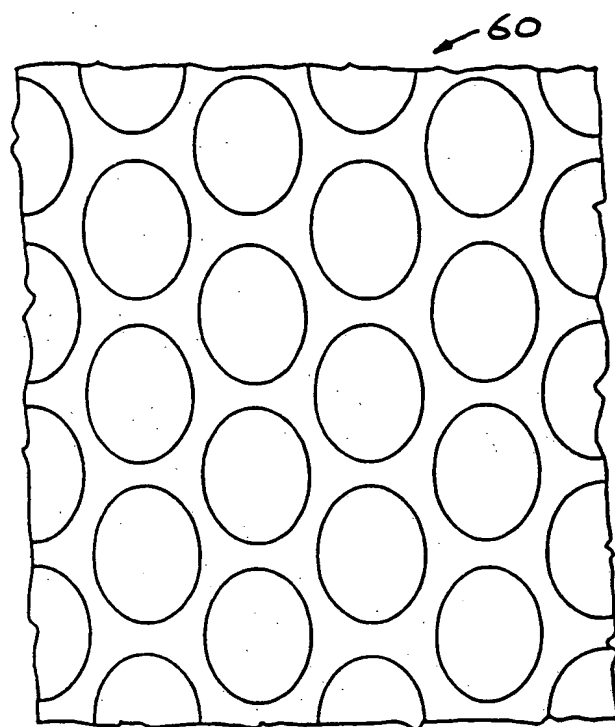


FIG. 7

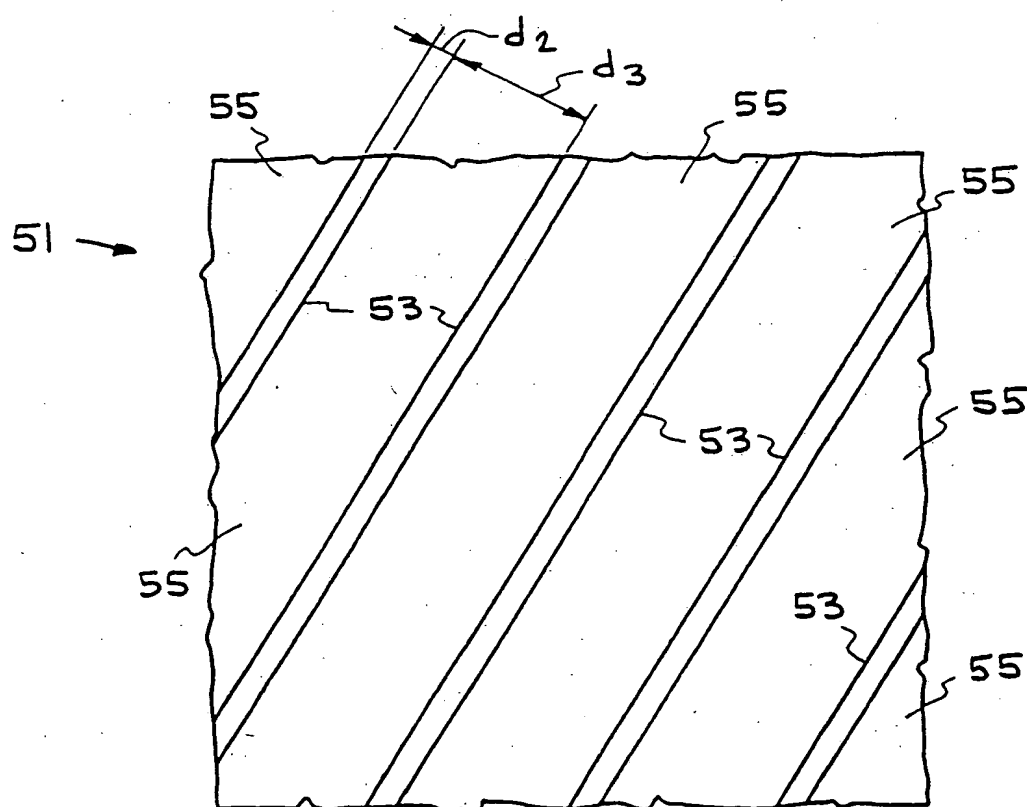
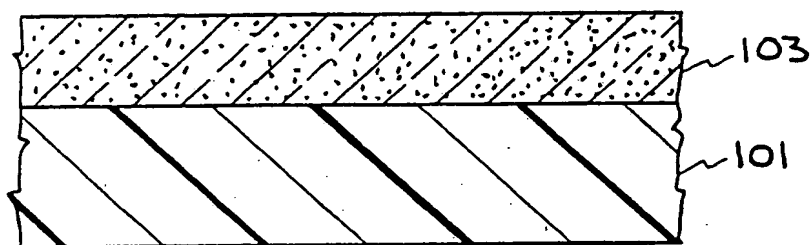
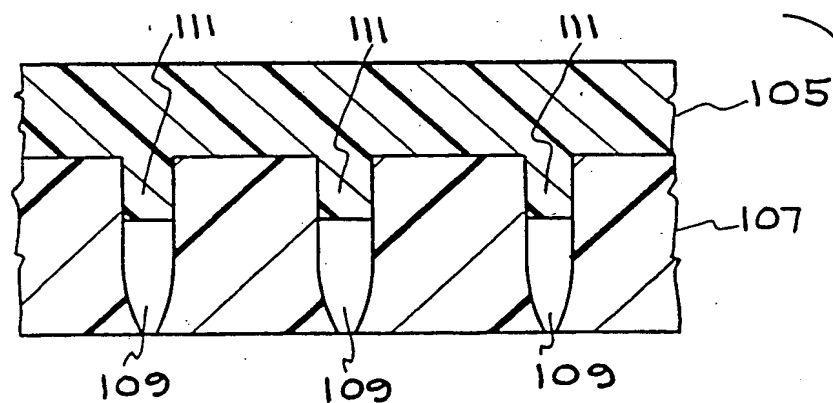
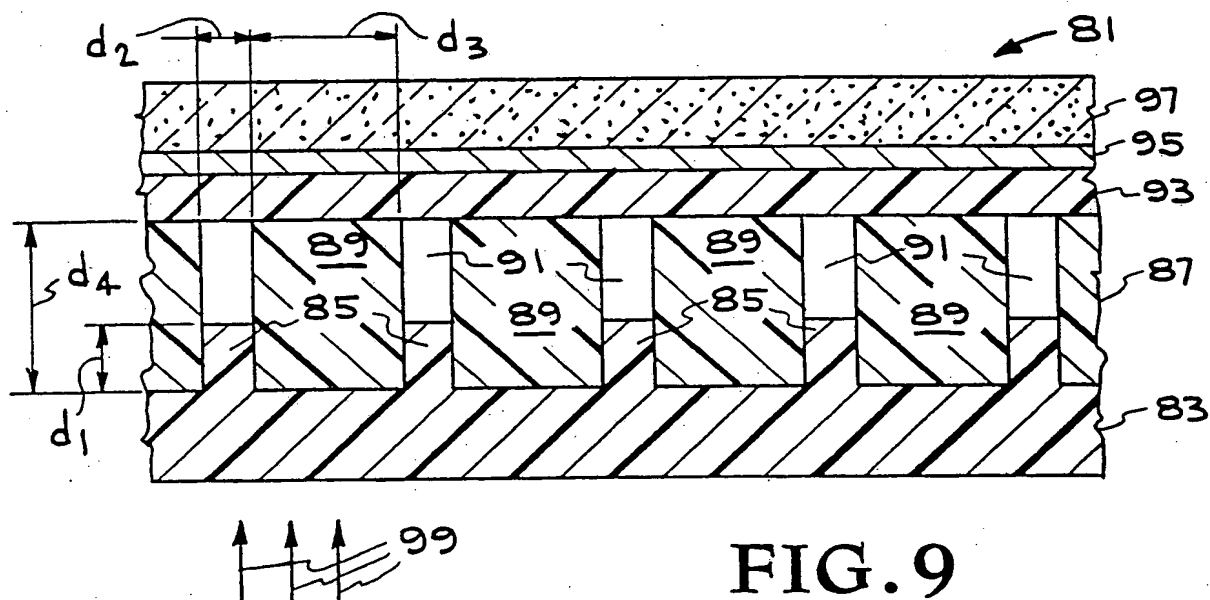


FIG. 8

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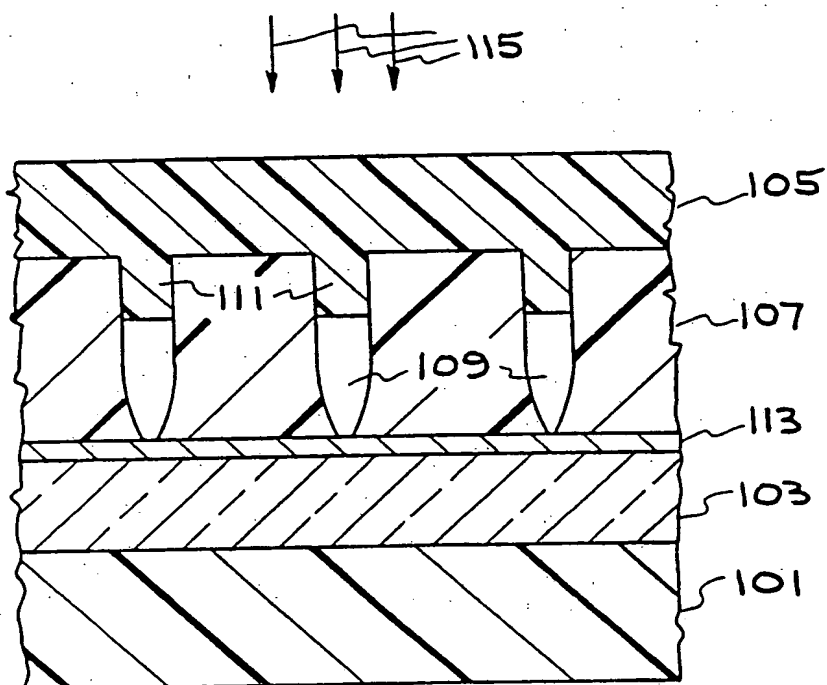


FIG. 10B

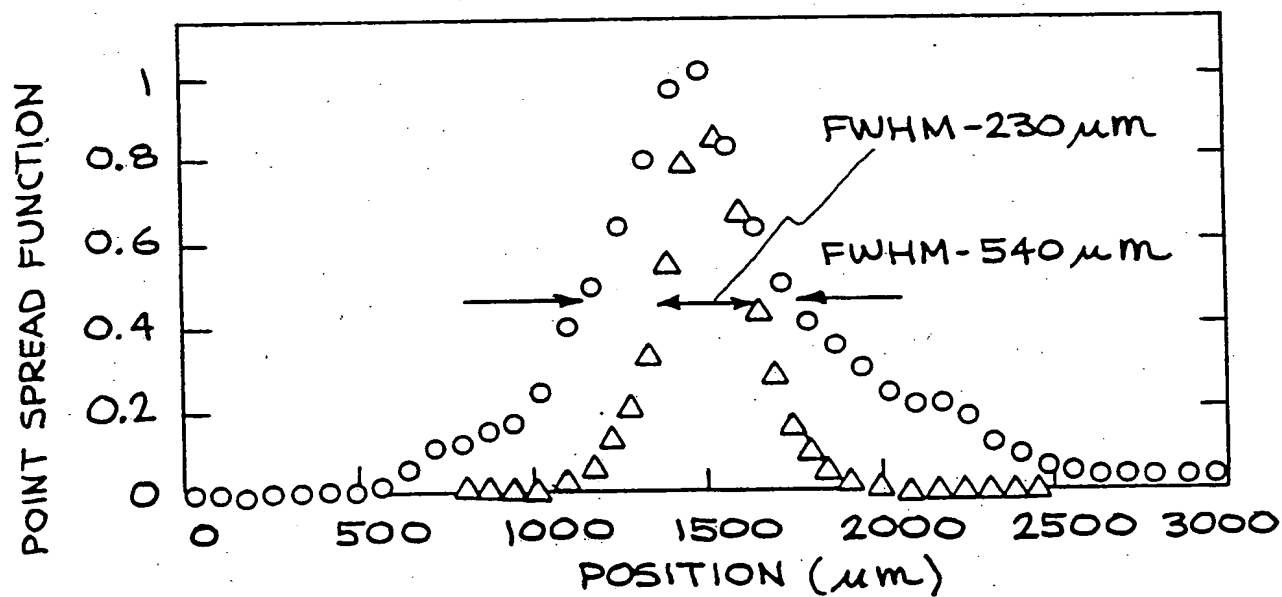


FIG. 11

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US92/06308

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(5) : H01L 21/306; B44C 1/22; B29C 37/00; C23F 1/00; G01T 1/20  
US CL : 156/655,668; 250/361R; 427/065

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 156/656,657,659.1,662; 250/367, 313/463; 427/064

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim
A	US,A, 3,783,297 (Houston) 01 January 1974 See col. 2, lines 19-47.	1-22
A	US,A, 4,069,355 (Lubowski et al) 17 January 1978 See Figures 2a-2h and 3a-3h.	1-22
A	US,A, 4,656,359 (Persyk et al) 07 April 1987 See col. 2, line 60-col. 3, line 28.	1-22

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search

15 SEPTEMBER 1992

Date of mailing of the international search report

17 NOV 1992

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